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Influence of the number of fatigue cycles on the peak shape of
X-ray rocking curves at duplex steel samples treated by VHCFA.K. Hüsecken^{a*}, M. Söker^b, K. Istomin^a, B. Dönges^c, H.-J. Christ^c, U. Krupp^b and U.
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Abstract

This paper shows results of in-situ X-ray diffraction analysis of VHCF cycles in duplex steel samples measured in reflection geometry at DELTA synchrotron. Due to the grain size a few number of grains inside the illuminated area fulfill the Bragg condition simultaneously and allow single grain analysis. Rocking curves (RC) recorded after $N \cdot 10^7$ fatigue cycles ($N=0 \dots 8$) with increasing stress load reveal changes of shape and intensity of selected austenite and ferrite grains as function of N and are interpreted by rotation of diffracting lattice plane due to formation of slip bands.

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Keywords: austenitic-ferritic duplex steel; VHCF; X-ray diffraction; single grain analysis; peak broadening; peak splitting

1. Introduction

Austenitic-ferritic duplex stainless steel has a high strength and an excellent corrosion resistance. Therefore it is used, for example, in offshore systems, where the material experiences cyclic loading of high stress amplitudes which may cause failure after long periodic operation. Laboratory experiment in very high cycle fatigue (VHCF) regime (up to 10^9 cycles) may help to evaluate the defect evolution as function of load cycles and to predict possible origins of crack formation [1]. Besides macroscopic probes TEM and X-ray techniques are important because with

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them it is possible to study the defect structure on the micro and nanoscale. Moreover, X-ray diffraction is a suitable technique to probe the defect formation during the fatigue treatment. Recently we have demonstrated that by use of micron sized beam and high energy photons X-ray diffraction allows single grain analysis of ferritic duplex steel samples after certain fatigue cycles. It has been indicated that X-ray Bragg peaks of austenite grains exhibit a certain probability of peak splitting which increases as a function of local stress load [2]. At the same time the shape of ferrite grains kept nearly not affected. Considering that this peak splitting can be used as indication of local stress impact, this technique has been applied for in-situ probing of the defect formation. It has been found that the probability of peak splitting increases with the number of fatigue cycles [3]. This paper will show the results of in-situ investigation of duplex steel samples with increasing stress load in reflection geometry. Because former fatigue experiments have shown, that the crack initiation predominately takes place at the surface of the investigated duplex steels [4], surface sensitive in-situ measurements were carried out in reflection mode using flat dog bone shaped sample. Here the probed information depth is in the order of 30 microns and allows for a closer comparison with other surface probe techniques.

2. Experimental details

The austenitic-ferritic duplex stainless steel has been produced accordingly to German standard 1.4462. Details of sample preparation are explained in [1]. After treatment the mean grain size was about 40-50 μm for ferrite and 30-40 μm for austenite (Fig. 1a).

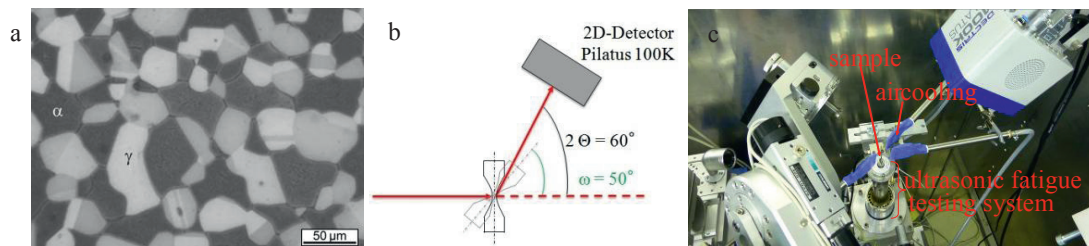


Fig. 1. (a) Austenite (fcc, bright) and Ferrite (bcc, dark) microstructure after heat treatment of the investigated duplex stainless steel [4]; (b) sketch of the experimental setup used at BL10; (c) experimental setup at BL 10

The in-situ X-ray diffraction was performed at beamline BL 10 at DELTA in Dortmund using a photon energy of 15 keV in reflection mode and a Pilatus detector with 195 x 487 pixels of 172 μm equipped in a distance of 251 mm behind the sample. The experimental scheme is shown in Fig. 1b. A beam with diameter of about 500 μm hits the specimen. Due to random orientation of grains with respect to the incident beam only a few grains fulfill the Bragg condition and produce diffraction spots on the detector, arranged in circles with opening angle 2θ with respect to the incident beam. Because of the small number of grains illuminated simultaneously each diffraction spot on the 2D detector (see Fig. 2) comes from a few grains only and allows for single grain analysis. At the same time the sample is equipped in an ultrasonic fatigue testing machine (UFTM) and can be probed after application of certain numbers of fatigue cycles. In order to measure changes in peaks widths the whole system (sample and UFTM) can be rotated around the horizontal axis to perform rocking scans. For the present experiment the rocking angle of the sample with respect to the incidence beam was changed between $-1^\circ < \omega < +1^\circ$ in steps of 0.04° . The RC displays several peaks which can be assigned to different grains showing different orientations with respect to the incident beam. In this experiment the stress amplitude was varied from 250 MPa up to 410 MPa in steps of 20 MPa and for each load value the sample was fatigued up to 10^7 cycles (see table 1). After each fatigue step the UFTM was stopped to measure the sample. The sample was destroyed after $8.175 \cdot 10^7$ cycles.

Table 1. Fatigue measurement parameters. After $8.175 \cdot 10^7$ cycles the sample broke.

Amplitude [MPa]	0	250	270	290	310	330	350	370	390	410
Load cycle	0	$1 \cdot 10^7$	$2 \cdot 10^7$	$3 \cdot 10^7$	$4 \cdot 10^7$	$5 \cdot 10^7$	$6 \cdot 10^7$	$7 \cdot 10^7$	$8 \cdot 10^7$	$8.175 \cdot 10^7$

3. Results and Discussion

Fig. 2 shows the 2D intensity distribution within the 2D detector before starting cyclic load. It shows several diffraction spots arranged on circles of fixed diffraction angle 2Θ . Depending on the respective d-values each ring can be indexed to either austenite or ferrite lattice. Along the rings several spots show up, numbered by boxes. Due to the large area of illumination the intensity distribution within the spot is not uniform and may refer to appearance of more than one grain (see insets of Fig. 2)

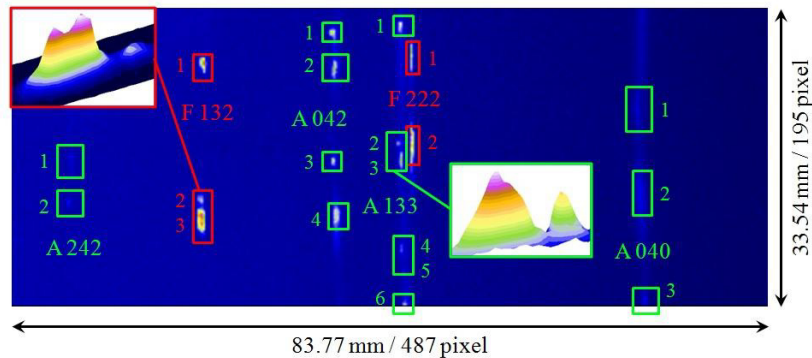


Fig. 2. 2D diffraction pattern measured during the in-situ experiment at DELTA synchrotron. Each reflection spot is arranged on a circle of fixed diffraction angle. The rings can be indexed to either austenite (A) or ferrite (F) lattice.

Due to the large pixels and relative short sample-detector distance, size and arrangement of the detector, spots remain nearly unchanged during fatigue cycles. An overlay of the 2D patterns taken after each load cycle provides marginal changes only. Higher visibility is obtained evaluating the RCs. Fig. 3 shows the evolution of a selected A042 grain as function of load cycles. The virgin sample shows a peak at $\Delta\omega = -0.5^\circ$ and a broad tail towards higher angles. After $N=2 \cdot 10^7$ cycles corresponding to a load of 270 MPa, this tail develops as a separate peak with an angular displacement of $\Delta\omega = 0.75^\circ$ with respect to the first one. The second peak probably belongs to a second grain which experiences a rotation during the fatigue treatment. The change of both peak intensities is shown in Fig. 3b. Except the RC of load cycle 3 ($N=3 \cdot 10^7$ and stress of 290 MPa), which may be caused by experimental uncertainties, the intensity of both peaks decrease in intensity up to $N=4 \cdot 10^7$ followed by a rather constant intensity of peak 1 but increase of peak 2. Remarkable is the strong increase of peak 2 close the destruction of the sample. Similar effects have been observed at grain of the austenite 133 reflection (not shown). The grain A133 consists of four peaks forming two groups separated by about $\Delta\omega = 1.5^\circ$. All four peaks show major changes in intensity and a slight shift of the peak position during the fatigue treatment. Whereas one peak stays almost unchanged all other peaks show remarkable increase or decrease of intensities revealing rearrangement of grains within the specimen during the fatigue treatment.

For the visibility of peak variations in the X-ray diffraction experiment three major conditions have to be considered: (1) the angle between direction of stress load and glide plane normal of the crystal lattice, (2) the angle between direction of stress load and the direction of gliding and (3) the projection angle of the peak splitting onto the incidence plane of X-ray experiment. The first two components are usually described by Schmid factor [5], the last one considers that using synchrotron radiation high resolution is achieved only in direction perpendicular to the electron orbit. In the present experiment the direction of stress load is always perpendicular to the electron orbit. In addition due to the use of a 2D detector with a small opening angle only those grains are measured where the normal to the diffracting lattice plane is nearly parallel to the incidence plane. However, the angle between glide direction and incident beam is random.

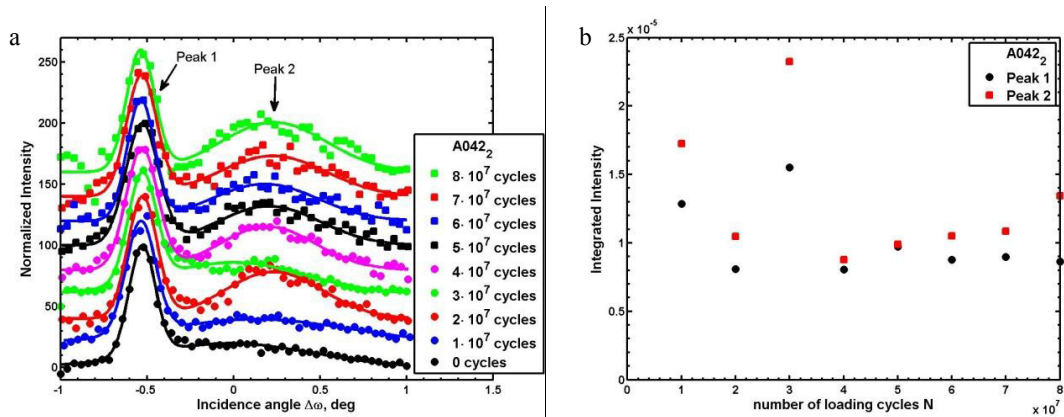


Fig. 3. (a) RCs show the evolution of the austenite 042 reflection as function of load cycles;
(b) intensity variation of both peaks as function of load cycles.

In the present experiment several grains stay unchanged because the respective glide direction is oblique to the incidence plane. However a few of the measured grains show major changes of shape and intensity, mainly a rotation by a few tenths of degree. This can be interpreted by rotation of lattice plane due to the formation of a dislocation network and subsequent formation of small grain boundaries. Considering the relation between dislocation density and the angle of forming grain boundary [6] the measured rotations indicate a change in the accumulated dislocation density. In contrast to previous experiments we found these changes not only in austenite but also in ferrite grains. Following current models one expects the formation of dislocation networks preferentially in austenite grains resulting in stress release in all grains. However, at present stage of data treatment, we cannot distinguish between grain rotation and strain impact from the diffraction pattern. This requires a new technical solution [7] as demonstrated recently.

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